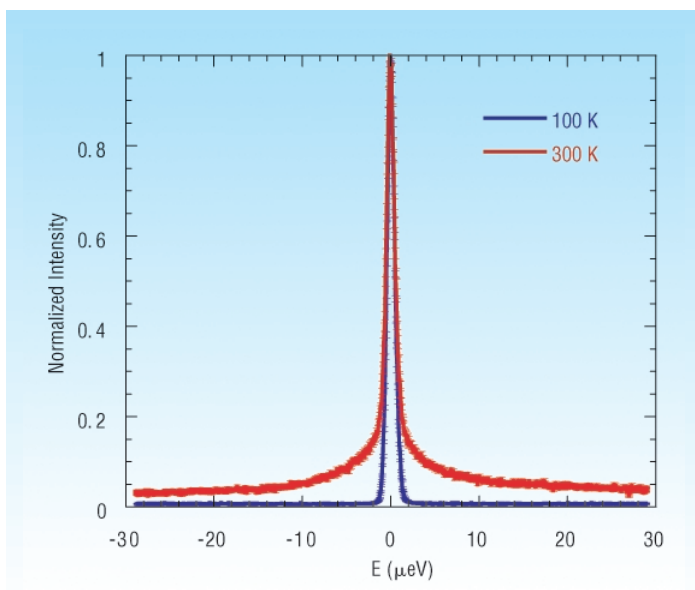


# INSTRUMENTATION DEVELOPMENTS

## IMPROVEMENTS TO THE PERFORMANCE OF HFBS

The recent commissioning of the high flux backscattering spectrometer (HFBS) has opened up opportunities to a variety of users from around the country to study the dynamics of condensed matter systems with time scales on the order of nanoseconds ( $10^{-9}$  s) and length scales up to  $10 \text{ \AA}$ . A careful design which incorporates state-of-the-art neutron optics has resulted in a flux on sample as high as that of any other backscattering spectrometer in the world with comparable energy resolution. The first call for proposals for the HFBS was made in FY 1999 and the response was outstanding. Several successful user experiments have already been performed (see Fig. 1) with more scheduled in the near future. Although the initial performance of the instrument was impressive, improvements continue to be made.

One of the recent changes to the instrument has been a modification to the data acquisition system. Initially the data were binned to the velocity of the reciprocating silicon monochromator. However the method in which the velocity was determined overemphasized local details of the monochromator motion which were not observed



**FIGURE 1.** Measurement of quasi-elastic scattering from monolayer coverage of alkanes on grafoil performed on the HFBS by H. Taub, D. Fuhrmann, L. Criswell, and K. Herwig. The low temperature data (blue) is resolution limited with a full-width at half maximum of  $0.96 \mu\text{eV}$ . The higher temperature data (red) clearly displays broadening indicative of the diffusive motion of the alkanes.

in the neutron data. A detailed analysis of the performance of the monochromator system indicated that the quality of the data could be improved by making a rather simple change in the way that data is collected and stored, namely by binning data versus time during a period of the Doppler monochromator motion. The data are later re-binned to velocity using the average monochromator motion profile. This straightforward change to the way that the data are collected has improved the quality of the data, suppressing the spurious effects observed in the velocity-binning mode.

Improvements have also been made to the Doppler monochromator system which have improved its reliability and performance. One of the problems encountered is associated with the fact that the monochromator is being driven to higher energy transfers than any other Doppler monochromator system in the world. The monochromator, which is vibrated over a distance of 9 cm at high frequencies, can experience accelerations in excess of  $100 \text{ g's}$ . Prolonged vibration of the monochromator resulted in the silicon wafers coming off of the monochromator surface. Recent changes in the gluing method have dramatically increased the lifetime of the silicon wafers on the monochromator.

Efforts to optimize shielding in various parts of the instrument have resulted in improvements in the signal to noise ratio in the detectors. During normal operation the instrument vessel (which is made up of the sample, analyzer crystals, and the detectors) is evacuated in order to reduce the background due to air scattering. Additional shielding has been installed on the detector assembly and on parts of the analyzer system in the vessel. Improvements have also been made to the shielding on the phase space transform chopper to handle the highly divergent beam from the converging neutron guide. This has resulted in a substantial increase of the signal to noise ratio to almost 600 to 1.

## THE DISK CHOPPER SPECTROMETER

The Disk Chopper Spectrometer (DCS) is an extremely versatile time-of-flight instrument, designed to achieve a broad range of energy resolution full widths, from  $12 \mu\text{eV}$  to  $1 \text{ meV}$ , by changing chopper speed, wavelength, and/or beam width. Once commissioned, the DCS will be in high demand for experiments on a variety of systems such as proteins, molecular crystals, disordered materials, and metal-hydrogen systems. The instrument is to be



**FIGURE 2. Photograph of the DCS detector bank and flight chamber.**

included, on a limited basis, in the first Call for Proposals in FY2000.

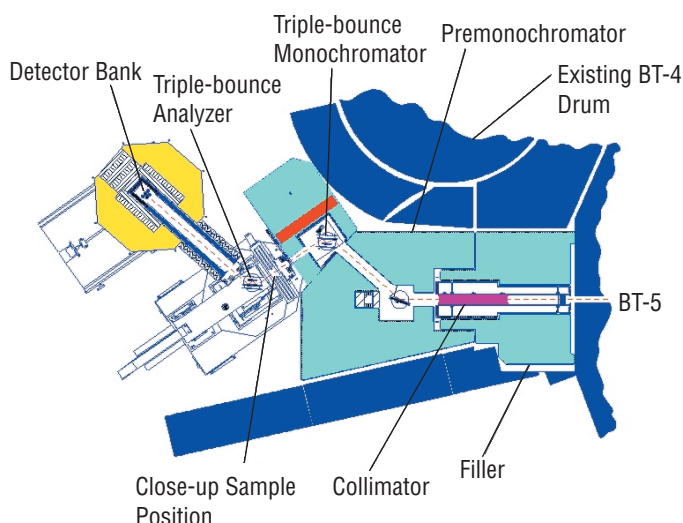
Over the past twelve months the DCS has seen major progress in a number of important areas. The sample chamber and flight chamber were equipped with beam-handling components, and the insides of both chambers were lined with cadmium. Flow tests were performed in order to complete the design of the gas handling system for the sample and flight chambers; the system had been fabricated and had largely been assembled by the end of the fiscal year. An overhead platform, to be used for sample environment preparation and installation into the sample chamber, has also been designed.

Problems with some of the amplifiers for the DCS detectors prompted us to send the complete inventory to the manufacturer for mutually agreed modifications. On their return discriminator thresholds were individually set, and the full complement of 913 detectors and amplifiers was installed on the detector racks, which were then installed and aligned at the spectrometer. The detectors and voltage distribution assemblies were cabled and fully tested prior to installation of the outer shields. Much work was devoted to troubleshooting and greatly improving the reliability and performance of the VME data acquisition electronics, and to the development of data acquisition software.

## PERFECT CRYSTAL SANS DIFFRACTOMETER

A perfect crystal diffractometer (PCD) used for very high resolution small angle neutron scattering (SANS) is being developed jointly by the NCNR and NSF as part of the CHRNS facility. The higher resolution obtained by using perfect silicon crystals increases the maximum size of features that can be measured from  $0.1\ \mu\text{m}$  obtained using the current NCNR's two 30 m, pinhole geometry SANS instruments, to  $10\ \mu\text{m}$  with the new instrument. The monochromator and analyzer use triple reflections before and after the sample. Using large channel-cut silicon crystals suppresses the "wings" of the beam profile, improving the signal-to-noise ratio to values comparable to that obtained from the pinhole instruments. The PCD will cover a Q-range from  $0.0004\ \text{nm}^{-1}$  to  $0.1\ \text{nm}^{-1}$  with an expected beam current of  $50,000\ \text{s}^{-1}$ .

Located on the BT-5 beam tube in the Confinement Building, the layout of the instrument is shown in Fig. 3. A vertically and horizontally focusing graphite premonochromator directs the neutron beam away from the main reactor beam towards the perfect crystal monochromator. The monochromator reflects a highly collimated beam to the sample. After the sample, a high precision rotation stage rotates the analyzer crystal to scan the small angle scattering obtained from the sample, which is then collected by the detector. The shields supporting the premonochromator are set on kinematic mounts for accurate repositioning. The monochromator and analyzer are isolated from room vibrations using a heavy pneumatic vibration isolation table.



**FIGURE 3. Schematic layout of the BT-5 perfect crystal diffractometer.**

In 1999, all detailed design work was finished and all purchased parts received. The beam shutter and premonochromator shielding have been installed. Major components yet to be installed are the detector housing, sample position table, and the small monochromator shield. Preliminary characterization measurements leading to instrument commissioning are planned for early 2000.

### THE FILTER ANALYZER NEUTRON SPECTROMETER

The new Filter Analyzer Neutron Spectrometer (FANS) is designed to give U.S. researchers access to unprecedented sensitivity for measuring the vibrational spectra of a wide variety of materials. This spectrometer, which will replace the current BT-4 instrument, uses cryogenically cooled polycrystals as low energy band-pass filters for neutrons scattered from the sample. A dramatic gain in signal over the existing filter analyzer is achieved primarily through a large increase in the solid angle covered by the secondary spectrometer. The FANS instrument is being developed in two phases. Phase I includes the first of two new filter assemblies, and Phase II includes the second filter analyzer and a new monochromator and monochromator drum system.

During the past year, the detailed design of the first filter wedge was completed, the vacuum chamber which encloses the Be and graphite filters was delivered, and the Be and graphite filter wedges were assembled (see Fig. 4). Recent tests demonstrated that these filters can be cooled to cryogenic temperatures which is necessary to maximize the performance of the instrument. Furthermore all of the parts for the undercarriage for Phase I have been received, assembled, and operated successfully under load. Most of the shielding and data acquisition system have also been received. Major assembly and installation of Phase I will begin early in 2000.

### DEVELOPMENT OF AN ACTIVE DOUBLE FOCUSING MONOCHROMATOR SYSTEM

The use of vertical or horizontal focussing monochromators to increase the intensity of selected neutrons incident on the sample is well known and widespread. Less common are systems combining these features. Often the machinery employed to do this is cumbersome and results in extraneous material in the beam, increasing the background from unwanted neutrons. For horizontal focussing it is desirable to have maximum flexibility by adjusting many

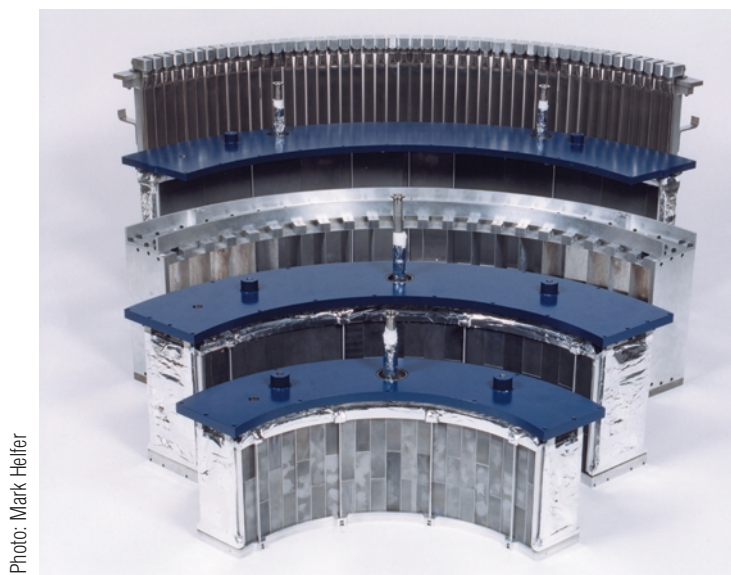
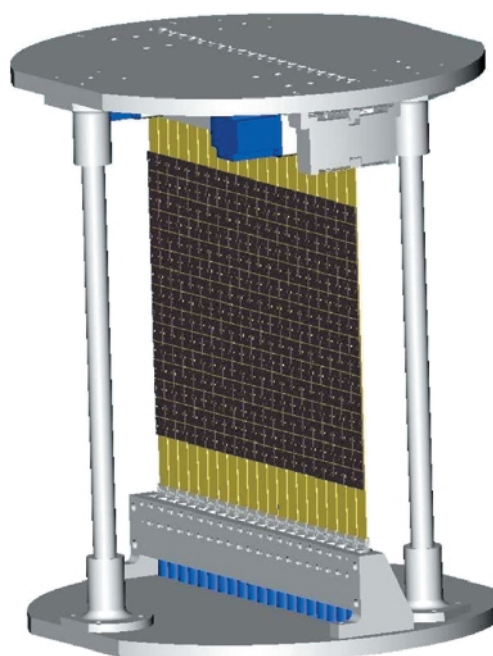


Photo: Mark Hefner

**FIGURE 4. The FANS filter assembly. The first Be filter is in the foreground with the PG filter second and the final Be filter in the background just before the detector bank. A radial collimator is visible in the middle of the assembly.**



**FIGURE 5. A drawing of the active double focussing monochromator system.**



monochromator elements while keeping the corresponding mechanical components away from the beam area. By contrast, vertical focussing can be confined to the problem of adjusting only one parameter, the radius of curvature of the array, and admits a correspondingly simpler mechanical solution.

We are currently developing a system that combines completely flexible horizontal focussing with vertical focussing that is achieved by buckling the entire system of separately controllable vertical crystal arrays. Cylindrical curvature is to be accomplished by compressing the system of specially shaped strips upon which the individual monochromating crystals are mounted. This design eliminates extraneous material in the beam, which should result in greatly reduced background.

### **AN ADVANCED LIQUID HYDROGEN COLD SOURCE**

The NIST liquid hydrogen cold source has completed over four years of service. It was installed with three goals: at least double the cold neutron intensity with respect to its predecessor ( $D_2O$  ice); operate simply and reliably; and pose no safety threat to the reactor or personnel. It has successfully met or exceeded all these goals. The cold neutron flux increased by a factor of 4 to 6, for wavelengths in the range of 0.2 nm to 2 nm. The availability of the source has been nearly 99 % of the time that the reactor was available (the reactor is shut down if the source is inoperable). And there have been no hydrogen leaks, nor have any of the insulating vacuums or helium containments been compromised.

Even as Unit 1 was being installed in 1995, however, improvements in the MCNP model (used for Monte Carlo simulations) of the NIST reactor were pointing toward a new, but more complicated cryostat assembly, with a possible additional gain of a factor of two. Better coupling between the reactor fuel and the cold source can be achieved by expanding the cooling jacket into the volume now occupied by the insulating vacuum, so that it partially surrounds the moderator vessel. In this way, the  $D_2O$  coolant also serves as an extension of the reactor reflector. Additional changes in the new source are based on our operating experience with the existing  $LH_2$  source and extensive MCNP calculations. Unit 2 will be an ellipsoidal shell rather than spherical, it will have an evacuated inner vessel, rather than vapor-filled, and the  $LH_2$  layer will be up



Photo: L. A. Shuman

**FIGURE 6. NCNR Mechanical Engineering Technician Scott Slifer welds the aluminum moderator vessel for the advanced hydrogen cold source.**

to 30 mm thick, rather than 20 mm, without increasing the  $LH_2$  volume.

The advanced liquid hydrogen cold source is currently being fabricated and will be installed in the NIST reactor next year. Enhanced mechanical design and manufacturing tools are exploited in the fabrication of the advanced source. Components of the hydrogen, insulating vacuum, helium containment, and  $D_2O$  vessels are cut from solid blocks of Al 6061 on a computer-controlled, high-speed mill at the NIST Instrumentation shop, and are then welded and thoroughly tested by NCNR staff (see Fig. 6). It is expected that the flux of cold neutrons will increase by a factor of 1.8.

### **IMPROVEMENTS TO NCNR SAMPLE ENVIRONMENT EQUIPMENT**

The sample environment equipment has seen a number of changes in FY1999 at the NCNR. One of the most visible accomplishments has been the development of informative webpages that detail sample environment resources, specifications, and even the current operating condition and location for specific devices. This information is easily accessible through the NCNR homepage (<http://www.ncnr.nist.gov/>), allowing guest researchers to plan their experiments and design appropriate sample holders.

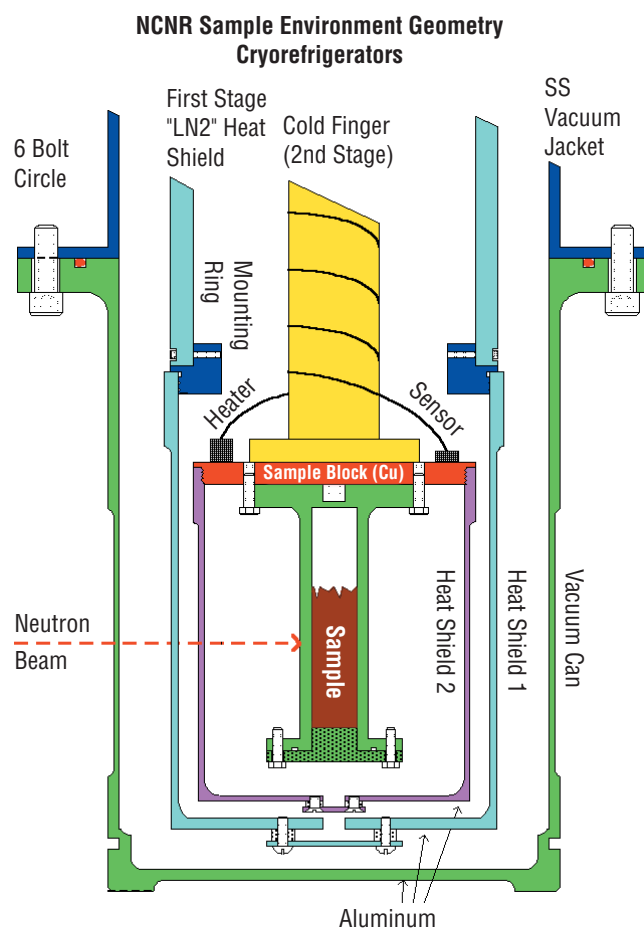
Commissioning has begun on a new high magnetic field/ low temperature superconducting magnet system which is financed through a joint collaboration of Johns Hopkins University, NCNR, NEC Research Institute, Princeton, the University of Maryland, and a National Science Foundation IMR grant. The 0.022 K dilution refrigerator of this new system has already been tested and used on triple axis spectrometers. A 7 T magnet has been temporarily outfitted in the system at this time, with replacement by a much stronger superconducting magnet of 10 T to 12 T in the coming months. The magnet is capable of operating with either a homogeneous magnetic field at the sample position or with a field gradient, a great aid for polarized neutron beam experiments. This system is

top loading, which provides an important capability for experiments requiring multiple sample changes or for quick changeover between consecutive experiments.

From new capabilities to improved sample preparation and screening, the sample environment resources have been significantly expanded during the past year. Listed in Table 1 are further acquisitions, notable modifications, and improvements that combine to offer the researcher more control over the sample conditions. Fig. 7 displays a typical cryorefrigerator sample environment. More details are available at the web site mentioned above.

**Table 1 Recent Sample Environment Acquisitions**

Helium flow cryostat	1.5 K to 300 K, dedicated to backscattering spectrometer
Helium flow cryostat	1.5 K to 300 K, dedicated to disk chopper time-of-flight spectrometer
Helium flow cryostat	1.5 K to 300 K
Pumped helium-3 cryostat insert	0.30 K to 300 K, for use with 7 T vertical field magnet
Closed-cycle helium refrigerator	7 K to 320 K
Closed-cycle helium refrigerator	10 K to 320 K, modified for backscattering spectrometer
Poiseuille flow shear cell	shear rate up to $130,000 \text{ s}^{-1}$ near surface
Light scattering particle sizer	2 nm to 100 nm diameter
Rheometer	$1.7 \times 10^{-3} \text{ Pa s}$ to $2.7 \times 10^8 \text{ Pa s}$ , $-60^\circ\text{C}$ to $600^\circ\text{C}$ , for in-situ measurement during SANS experiments
Lyophilizer	freeze dryer for sample preparation
Kare Fisher titrator	quantitative analysis of water in a sample
Freezer	$-20^\circ\text{C}$ , for longer term storage of biological samples
Superconducting magnet power supply	0 A to 120 A, bi-directional
Dual channel lock-in amplifier	1 mHz to 102 kHz frequency range
Four temperature controllers	programmable, remote operation
Dual channel lock-in amplifier	0 A to 120 A, bi-directional
Four temperature controllers	programmable, remote operation
Single-crystal windowed tail	for SANS low-temperature electromagnet experiments
Sensitive equipment transport carts	custom-designed to protect valuable equipment



**FIGURE 7. Schematic diagram of the sample environment in a cryorefrigerator in the neutron beam.**